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## DEFENCE SCIENCE AND TECHNOLOGY ORGANISATION MATERIALS RESEARCH LABORATORIES

MELBOURNE, VICTORIA

**TECHNICAL NOTE** 

MRL-TN-486

HIGH-PRESSURE OPTICAL CELL

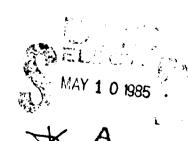
R. McLeary & A.J. Hutchins

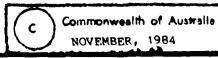
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R. McLeary & A.J. Hutchins

#### ABSTRACT

This technical note describes a high-pressure optical cell and associated pressurising equipment which have been constructed at MRL. The system provides gas mixtures in the cell at pressures of up to 200 MPa at temperatures in the range 200-300 K. The apparatus was required for an experimental investigation of collision-induced fluorescence and optical gain in optically-pumped gas mixtures at high pressure.

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#### HIGH-PRESSURE OPTICAL CELL

#### 1. INTRODUCTION

This technical note provides constructional details of a high-pressure cell and associated pressurising equipment which have been constructed at MRL. This apparatus was required for an experimental program investigating collision-induced fluorescence and optical gain in optically-pumped gas mixtures at high pressure [1], [2].

The experiments required a gas cell with two windows each transparent in the infrared regions 2.5  $\mu m$  to 3  $\mu m$  and 3.6  $\mu m$  to 5  $\mu m$ . The gases introduced into the cell were mixtures of deuterium (D<sub>2</sub>), argon (Ar) and hydrogen deuteride (HD). The maximum required operating pressure was 200 MPa at temperatures in the range 200-300 K.

A schematic diagram of the high-pressure system is shown in Figure 1. The major elements of this apparatus will be described in the following sections.

#### 2. HIGH-PRESSURES CELL

A number of cells of different geometries were constructed; a typical example is shown in Figure 2. Sapphire windows (thickness 6 mm), polished flat and parallel, were used in all cells. Sapphire has the required transmission [3] and strength properties [4] for this application. A small quantity of "glyptol" adhesive (a medium oil alkyd varnish) was used to seal the windows to the cell body. The cell body, clamping bolts and nuts were all stainless steel, while the window holders were made from monel metal. A polytetrafluoroethylene (teflon) washer was placed between the window and the window holder to protect the sapphire surface. The window holders were required to maintain a load on the window seal when the cell was evacuated prior to filling with the gas mixture.

For room-temperature operation, high-pressure (90 Durometer BL9013) O-rings were used to seal the cell components, Figure 3a. These O-rings provide an excellent seal and may be used a number of times. However, they are not suitable for operation at reduced temperatures, and in this case the alternative sealing arrangement, shown in Figure 3b, was used. The gaskets shown in this figure were constructed from oxygen-free copper (un-annealed) which was lightly covered with silicone rubber. This arrangement maintained the high-pressure seal at temperatures at least as low as 200 K, where experiments were conducted. This arrangement has the disadvantage that the copper gaskets can only be used once.

A subsidiary pair of windows was required at low temperature to prevent condensation of atmospheric water vapour on the cell windows. The arrangement used is shown in Figure 4. A convenient subsidiary-window material was mylar (0.05 mm thick), mounted on an electrically-heated aluminium plate which was attached to the cell by an insulating cone. The cone was sealed to the cell by a teflon 0-ring so that the volume between the mylar and sapphire windows could be evacuated. This system prevented condensation on all windows. An optical transmission curve for the mylar-window material is shown in Figure 5.

A simple bath containing alcohol and dry ice was used to cool the cell.

#### 3. GAS INTENSIFIER

The gas intensifier shown in Figure 6 is a simple piston/cylinder arrangement which was used to compress gas from a pressure of approximately 13 MPa up to a maximum pressure of 200 MPa. The intensifier was operated by means of a hydraulic press with a maximum load capability of 130 kN. The materials chosen for the components of the intensifier and relevant dimensions are indicated in Figure 6.

The relative volumes of the intensifier and the cell (plus connecting tuping) are such that a maximum gas pressure of 200 MPa was achieved only for initial cylinder pressures above 13 MPa. A full cylinder (size G) of argon, as received from the supplier, contains gas at a pressure of approximately 15.5 MPa. Thus only about one sixth of the gas from the cylinder could be utilized, when single-stage compression was used. However, this was not a serious problem, as only two cylinders of argon were required during the two-year experimental program.

Deuterium is normally supplied in cylinders at a pressure of approximately 5 MPa. Thus pure deuterium could not be compressed to 200 MPa with the intensifier alone. For  $D_2$ :Ar ratios of 5:8 or less, the intensifier by itself could adequately compress the gas to the required 200 MPa. However, for mixtures which were richer in deuterium, an auxiliary compression stage was required. This compression stage is labelled pre-compressor in Figure 1 and will be described in the next section.

#### 4. PRE-COMPRESSOR

The pre-compressor shown in Figure 7 consists of a high-pressure steel tube which contains a solid brass bobbin free to slide within the tube. The bobbin has a circumferential O-ring which seals on the bore of the tube and a teflon guiding ring. The end faces of the bobbin also contain O-rings which are able to seal either the entrance or exit ports in the end sections of the tube.

In operation the system was evacuated and then filled with gas mixture at a pre-determined pressure through the exit port (see Figure 1). This ensured that the bobbin was initially located at the entrance-port end of the tube. High-pressure argon (15.5 MPa) was then directed in through the entrance port, the bobbin was driven the length of the tube and the gas mixture was thereby compressed into the intensifier. A factor of slightly over three pre-compression was achieved in this way. This allowed the compression by the intensifier of all gas mixtures (including pure  $D_2$ ) up to a final pressure of 200 MPa. It also allowed more complete usage of the contents of the gas cylinders.

An experiment was undertaken to ascertain if there was any significant leakage of driver gas across the bobbin during compression of the gas mixture. In the experiment, helium was used instead of argon as the driver gas, and a sample of compressed gas was passed through a helium-detection apparatus. This apparatus could detect helium concentrations of less than one percent. Helium was not detected in the compressed gas.

#### 5. TAPS AND FITTINGS

"Superpressure" (Aminco) high-pressure taps, tees, crosses and connecting tubing rated at a working pressure of 200 MPa or above were used.

#### 6. CONCLUSION

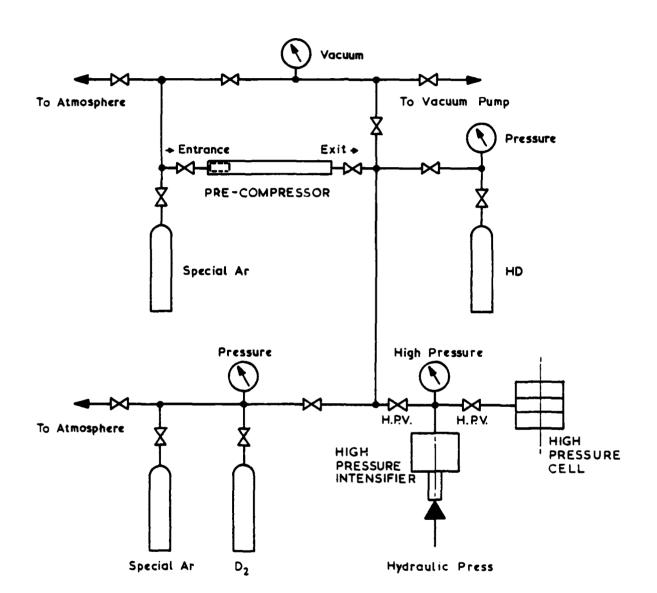
The main features of a high-pressure optical cell and associated pressurising equipment have been described. This apparatus has been used to investigate collision-induced-dipole effects in gas mixtures at pressures up to 200 MPa over a temperature range of 200-300 K.

#### 7. ACKNOWLEDGEMENT

The authors are grateful to Dr C.W. Weaver for helpful advice on components and materials for high-pressure applications.

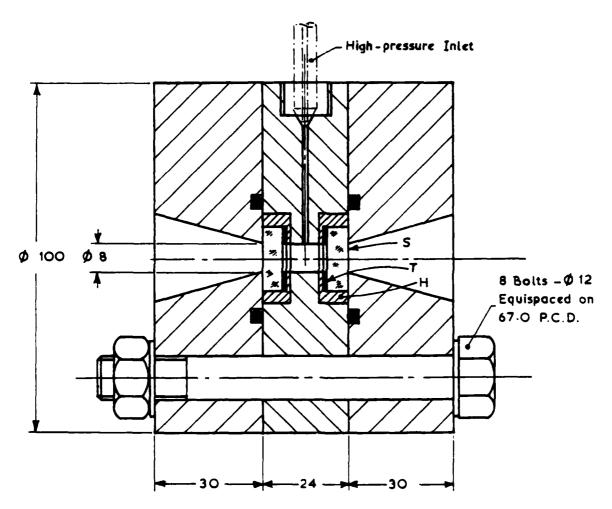
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- McLeary, R. "Measurement of Gain in an Optically-Excited Mixture of D<sub>2</sub> and Ar at High Density". To be published.
- 3. Oppenheim, U.P. and Even, U. (1962). "Infrared Properties of Sapphire at Elevated Temperatures". J. Opt. Soc. Am., 52, (9), 1078-1079.
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H.P.V.:- High Pressure Valve ('Aminco')

FIGURE 1. Schematic diagram of high-pressure system.



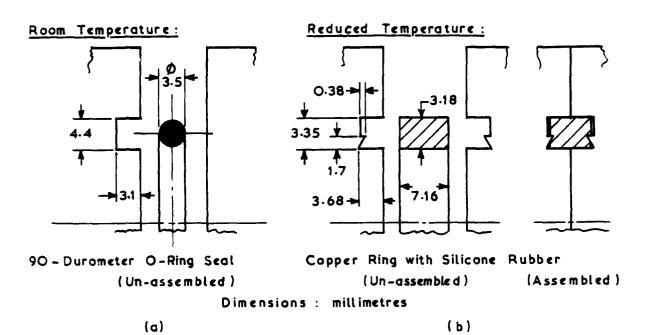
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S :- Sapphire Window

T := P.T.F.E. ('Tetlon') Washer

H :- Holder for Window Dimensions : millimetres

FIGURE 2. Schematic diagram of high-pressure optical cell.



- FIGURE 3. a. Sealing arrangement for room-temperature operation.
  - b. Sealing arrangement for reduced-temperature operation.

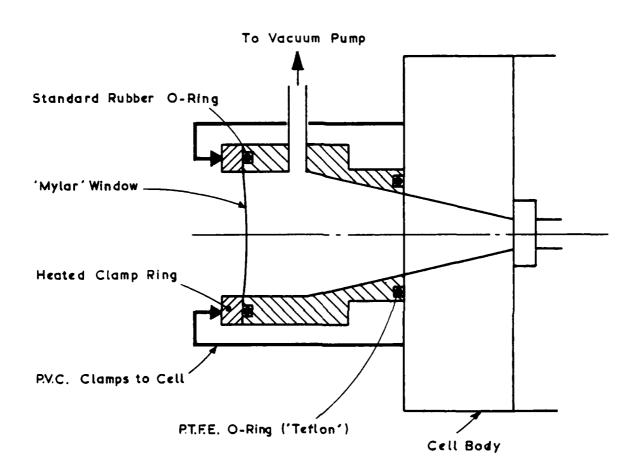
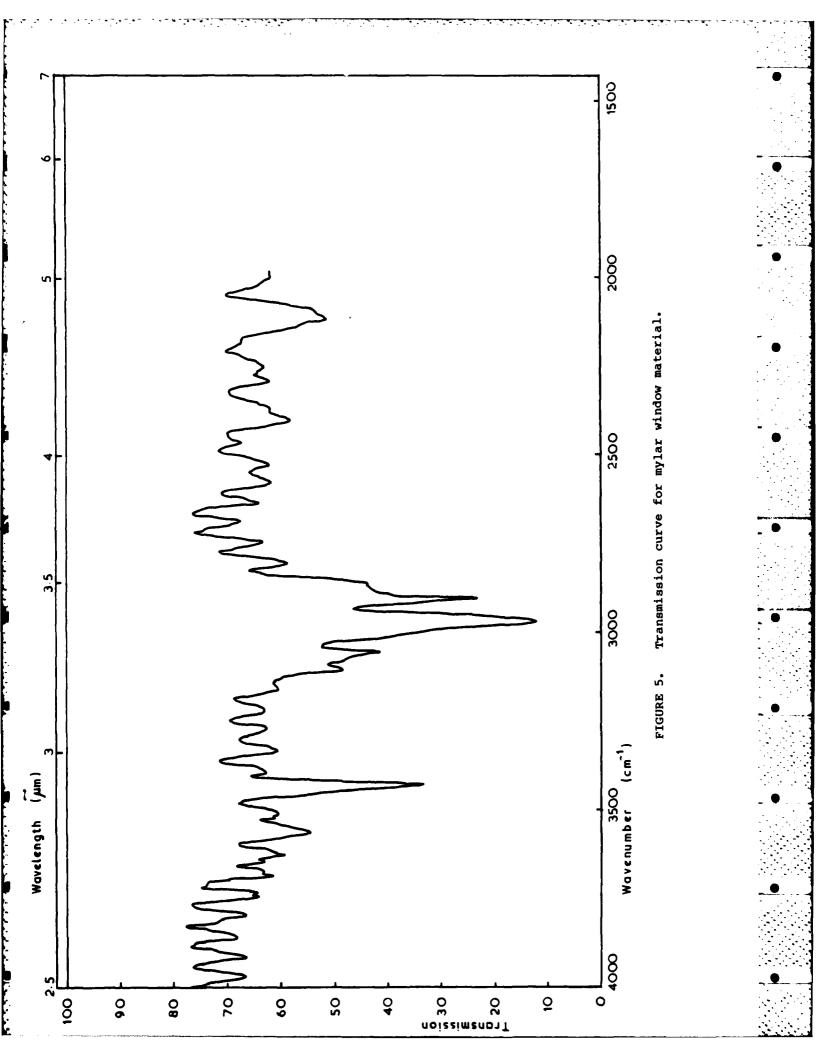


FIGURE 4. Schematic diagram of subsidiary-window arrangement.



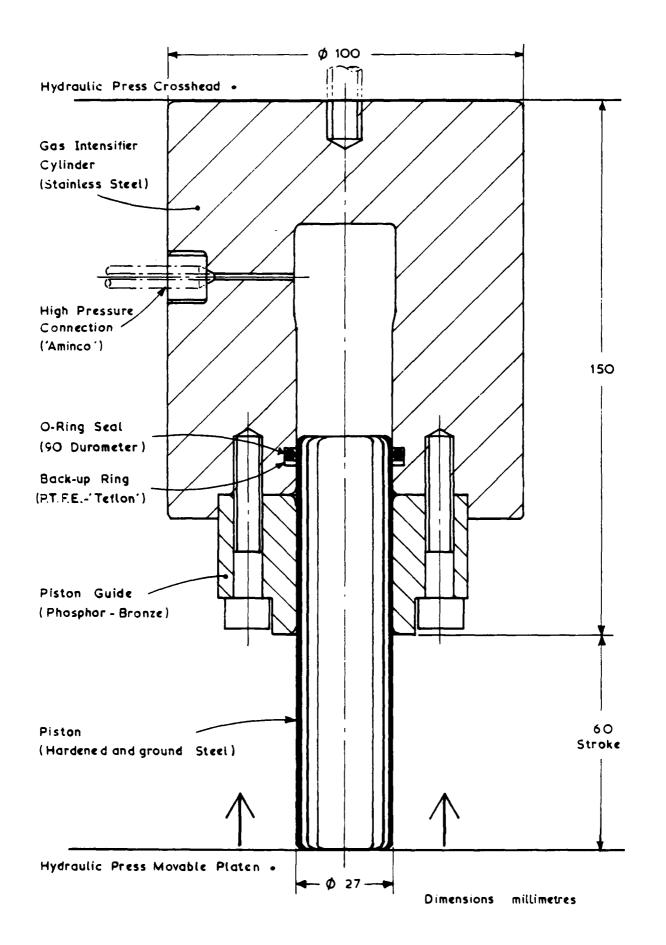


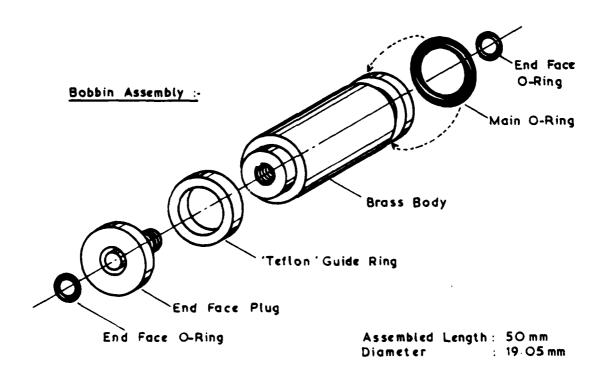
FIGURE 6. Schematic diagram of high-pressure gas intensifier.

Entrance Part End shown :

Exit Port End similar 
Sliding High Pressure Tube - Length 600 mm

'Aminco' High Pressure Tube - Length 600 mm

'Ferulok' Tube Fittings



# END

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